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## A NEW MODIFICATION OF SILICON CARBIDE WITH A RHOMBIC LATTICE

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Data of an x-ray structural study of single crystals of silicon carbide  $\alpha$ -SiC(6H) and a new, previously unknown modification  $\gamma$ -SiC with a rhombic lattice are described. The rhombic-lattice parameters are related to the parameter  $a$  of the hexagonal and cubic lattices of SiC in the following way:  $a_r = a_h$ ,  $b_r = 3a_h$ ,  $c_r = 3a_c$ .

Research on the crystalline forms (exterior symmetry) of silicon carbide using optical methods showed that this compound crystallizes in two systems: cubic and hexagonal [1]. This was subsequently corroborated in deciphering the crystalline structures of SiC using diffraction methods.

In doing so, it became possible to discover for the first time the monomeric form of polymorphism, namely, polytypism [2, 3]. The hexagonal modifications of SiC have either a primitive  $P$  cell or an  $R$  cell doubly centered by volume. The SiC phase with a face-centered cubic lattice ( $a = 4.3585$  Å) was denoted  $\beta$ -SiC, and the modification with a hexagonal or rhombohedral lattice was denoted  $\alpha$ -SiC.

The  $\beta$ -SiC modification has the structure of zinc blende (sphalerite); the sphalerite lattice parameter is  $a = 5.420$  Å, and the Fedorov symmetry group is  $F\bar{4}3m$ . In this group, like the  $F$ -symmorphous group, the minimum multiplicity of the set of equivalent positions is equal to 4. The atoms of zinc and sulfur occupy the following positions: for Zn,  $4(a) - 000$ ; for S,  $4(c) - 1/4, 1/4, 1/4$ .

There are four molecules per elementary cell. The structure consists of tetrahedrons of  $ZnS_4$  or  $SZn_4$ . Each vertex is common to 4 tetrahedra. The tetrahedra are gathered in layers parallel to each other, so that their faces are parallel to the lattice planes (111). In accordance with the requirements of densest packing, the layer sequence ABCABC... is formed along the direction [111]. The whole structure has the tetrahedral symmetry ( $43m$ ), and not the cubic symmetry ( $m3m$ ). In the first coordination sphere, a zinc atom is surrounded by sulfur atoms at the vertices of a tetrahedron, and in the second sphere, it is surrounded by zinc atoms arranged at the vertices of a cube-octahedron.

At a temperature of 1175°C, sphalerite transforms into wurtzite. Compared to zinc blende, the triplets of tetrahedra

in the wurtzite structure are mutually turned by 60° around their axes [111]. The tetrahedra are coupled in a three-dimensional skeleton by their common vertices. An octahedral interstice appears between each pair of triplets of tetrahedra. The columns of octahedral interstices arranged one over another form hollow channels. The spiral axis  $6_3$  passes through the centers of these channels. All identical tetrahedra face the same direction along this axis. A zinc atom in the first coordination sphere is surrounded by sulfur atoms at the vertices of a tetrahedron, and in the second sphere by zinc atoms arranged at the vertices of an anticubooctahedron. The elementary cell of wurtzite has the following parameters:  $a = 3.811$  Å,  $c = 6.234$  Å,  $c/a = 1.636$ . The axis ratio  $c/a$  virtually coincides with the "perfect" value, equal to 1.633, which characterizes the densest packing of spheres by the pattern ABABAB .... The Fedorov group is  $P6_3mc$ . The zinc and sulfur atoms occupy equivalent single-variant double positions  $2(b)$ :  $2/3, 1/3, Z$  and  $1/3, 2/3, 1/2 + Z$  ( $Z = 0.375$ ), and there are two molecules per elementary cell.

In compounds crystallizing in the wurtzite structure with an axis ratio  $c/a$  approaching the perfect ratio and with the coordinate  $Z = 0.375$ , each atom will be surrounded by a nearly right tetrahedron formed by atoms of another type. The relationships between the structures of zinc blende and wurtzite are the same as between the cubic and hexagonal densest packings. Modifications of silicon carbide have densely packed structures. Therefore, the possible Fedorov symmetry groups are limited: the densest packings of spheres belong to eight Fedorov groups. If the sphere is replaced by an asymmetric pair of atoms of silicon and carbon, then in the case of mutually tetrahedral arrangement of atoms, a number of symmetry elements will disappear. In conformity with this, SiC can belong to just four Fedorov groups:  $P3m1$ ,  $P\bar{3}m1$ ,  $P6_3mc$ ,  $F\bar{4}3m$  [2, 3].

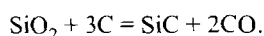
Sometimes, mixed packings of tetrahedra are formed in crystallization of silicon carbide, which are intermediate between densely packed cubic and hexagonal packings.

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Silicon carbide with the wurtzite structure ( $a = 3.0763 \text{ \AA}$ ,  $c = 5.0480 \text{ \AA}$ ,  $c/a = 1.641$ , Fedorov group  $P6_3mc$ ) was identified in the products of pyrolysis (hydrogen atmosphere) of mixtures of  $\text{SiCl}_4 - \text{C}_6\text{H}_5\text{CH}_3$  and  $\text{SiCl}_4 - \text{CH}_3\text{SiCl}_3$  at temperatures of  $1400 - 1500^\circ\text{C}$  [4]. In [5] light blue crystals in the form of dendrite needles consisting of well-faceted hexagonal prisms topped by similar pyramids were identified in the products of annealing (vacuum, temperature over  $1800^\circ\text{C}$ ) of a mixture consisting of carbon and aluminum and silicon oxides. Their position in the crucible indicated that they had crystallized from the gaseous phase. A microchemical analysis identified the presence of aluminum in the crystals. An x-ray phase analysis showed that they belong to a hexagonal system:  $a = 3.10 \text{ \AA}$ ,  $c = 4.99 \text{ \AA}$ ,  $c/a = 1.61$ . Indexing of the x-ray pattern of pulverized crystals made it possible to attribute them to the Fedorov groups  $P6_3/mmc$  and  $P6_3mc$ . The elementary-cell parameters of the light blue crystals virtually coincide with  $\text{AlN}$  and  $\alpha\text{-SiC}(2H)$ .

Analysis of the crystals by the Kjeldahl method to detect the presence of nitrogen showed that these crystals were not  $\text{AlN}$ . Pulverized crystals did not react with water or sulfuric acid either in the cold or in protracted boiling. They reacted slowly with alkali-metal hydroxide. The authors of [5] put forward the hypothesis that the light blue crystals are aluminum oxide  $\text{Al}_2\text{O}_3$ . However, this assumption contradicts the x-ray data presented in [5]. According to calculation, a hexagonal elementary cell of  $\text{Al}_2\text{O}_3$  corresponds to one formula unit, which is half the minimum multiplicity of the sets of equivalent positions of atoms in the Fedorov groups  $P6_3/mmc$  and  $P6_3mc$ .

Non-doped  $\beta\text{-SiC}$  is of yellow color, and  $\alpha\text{-SiC}$  is clear [6].  $\alpha\text{-SiC}$  doped with nitrogen and phosphorus acquires a green color, doped with aluminum it becomes light blue and black, and with boron it becomes brown and black. Assuming that the light blue crystals are silicon carbide, the calculated density for  $Z = 2$  is equal to  $3.206 \text{ g/cm}^3$ . This value coincides with the density  $3.214 \text{ g/cm}^3$  of  $\alpha\text{-SiC}(2H)$  and  $3.210 \text{ g/cm}^3$  of  $\beta\text{-SiC}$  [6]. Similar to silicon carbide, the light blue crystals decompose when fused with alkali-metal hydroxide [2, 5]. It is probable that in the study described in [5] crystals of  $\alpha\text{-SiC}(2H)$  were grown for the first time according to the reaction [2, 6]



The  $\alpha\text{-SiC}$  modification, besides the phase  $\text{SiC}(2H)$ , was identified in several dozen polytypal modifications [1 – 3, 6]. Those of them that are most frequently found in industrial samples are:  $\text{SiC}(4H)$  with  $a = 3.076 \text{ \AA}$ ,  $c = 10.046 \text{ \AA}$ , Fedorov group  $P6_3mc$ ;  $\text{SiC}(6H)$ ,  $a = 3.081 \text{ \AA}$ ,  $c = 15.117 \text{ \AA}$ , Fedorov group  $P6_3mc$ ; and  $\text{SiC}(15R)$ ,  $a = 3.073 \text{ \AA}$ ,  $c = 37.30 \text{ \AA}$ , Fedorov group  $R3m$  [2, 3, 6].

The large variety of  $\alpha\text{-SiC}$  polytypes and the preservation of long-range order in them can best be accounted for by

the mechanism of spiral growth through screw dislocations. Spiral growth patterns are most frequently observed at the face (001) [2]. However, this mechanism does not account for the formation of the polytypes  $\alpha\text{-SiC}(2H)$  and  $\alpha\text{-SiC}(8H)$ ,  $a = 3.079 \text{ \AA}$ ,  $c = 20.147 \text{ \AA}$ , Fedorov group  $P6_3mc$ , and some others [3].

Polytypal structures often interpenetrate in parallel growth along the axis  $c$ . Crystals taking part in twinning can have identical or different structures. Interpenetration of two or more structures can lead to formation of seemingly single crystals that have a different polytypal structure at different sites. A single crystal described in the literature had two different coexistent structures of the polytype  $36H$  in the upper and lower parts of it with identical hexagonal lattice parameters ( $a = 3.078 \text{ \AA}$ ,  $c = 90.65 \text{ \AA}$ , Fedorov group  $P3m$ ). The structures  $36Ha$  and  $36Hb$  differed by the arrangement of the layers within the limits of an elementary cell [2].

In all hexagonal polytypes of  $\alpha\text{-SiC}$ , the parameter  $a$  of the elementary cell is equal to  $3.078 \text{ \AA}$ . The parameter  $c$  is a multiple of  $2.518 \text{ \AA}$ . Thus,  $a = 3.078 \text{ \AA}$ ,  $c = n \cdot 2.518 \text{ \AA}$ , and the ratio  $c/a = n \cdot 0.817$ . The ratio  $c/a$  is nearly half the value  $n \cdot 1.633$ .

In the case of the densest cubic (face-centered) packing, the elementary-cell parameter  $a = 3h$  ( $h$  is the distance between neighboring layers of atom). With  $h = 2.518 \text{ \AA}$ , the parameter  $a = 4.362 \text{ \AA}$ . This value virtually coincides with the x-ray data for  $\beta\text{-SiC}$ :  $a = 4.3585 \text{ \AA}$ .

The cubic modification  $\beta\text{-SiC}$  is formed very rapidly in annealing of  $\text{SiO}_2$  and C. The modification  $\beta\text{-SiC}$  starts crystallizing at a temperature of  $1400 - 1500^\circ\text{C}$ , and at  $1700^\circ\text{C}$  the reaction ends. There is no definite temperature of the transformation  $\beta\text{-SiC} \rightarrow \alpha\text{-SiC}$ ; according to some data,  $\beta\text{-SiC}$  irreversibly transforms into  $\alpha\text{-SiC}$  at a temperature above  $2000^\circ\text{C}$ , and according to other data, it remains stable up to  $2830^\circ\text{C}$ .

It is assumed that the transformation  $\beta\text{-SiC} \rightarrow \alpha\text{-SiC}$  occurs at a temperature above  $2000^\circ\text{C}$  due to surface diffusion during recrystallization. It is established that the polytype  $6H$  prevails in the recrystallization products, and the second phase is the polytype  $15R$ .

Nitrogen stabilizes  $\beta\text{-SiC}$ . Several investigations suggest that  $\beta\text{-SiC}$  is not a low-temperature modification; it is metastable at all temperatures [2, 6].

The authors of [8] reported conversion of silicon carbide from the hexagonal to the cubic modification under the effect of high pressures and temperatures. The studies were carried out on a high-pressure/high-temperature stand. The sample was placed in a graphite capsule in a compression chamber and subjected to a pressure of  $3 - 7 \text{ GPa}$  at a temperature of  $1200 - 1400^\circ\text{C}$  for several minutes. An x-ray analysis was carried out at atmospheric pressure. The Debye crystallograms of all the samples exhibited a sharp decrease in the intensity of just the  $\alpha\text{-SiC}$  lines. The main lines of silicon carbide common to all its modifications did not change.

Compression of  $\alpha$ -SiC at room temperature did not lead to a change in the x-ray pattern of the samples. Later the authors of [8] once more investigated silicon carbide subjected to 1.5 – 7.0 GPa pressure at a temperature of 1200 – 1700°C [9]. In the study described in [9] the previously discovered phase transformation  $\alpha$ -SiC  $\rightarrow$   $\beta$ -SiC was confirmed. The transition  $\alpha$ -SiC  $\rightarrow$   $\beta$ -SiC occurs independently of the chemical purity and monophase character of the samples, in which  $\alpha$ -SiC is the main phase. The effect of high temperature (1200 – 2200°C) and pressure on the phase transformation  $\alpha$ -SiC  $\rightleftharpoons$   $\beta$ -SiC was investigated in [10] using the method of high-temperature x-ray analysis.

In the study in [10], transformation of the hexagonal modification to the cubic one was not observed either in the presence of impurities or with an excess of silicon. It was found that boron nitride facilitates the formation of  $\beta$ -SiC, whereas iron impurity facilitates the transformation of  $\beta$ -SiC to the  $\alpha$ -SiC modification.

A phase transformation was observed in silicon carbide under shock waves at a pressure of 24 GPa [11] and in a high-pressure unit at 30 GPa [12]. According to the data in [12], this transformation is related to the transition to the metallic phase. The study in [13] investigated the change in the compressibility of  $\beta$ -SiC with change in the pressure at room temperature. It was found that the compressibility of  $\beta$ -SiC depends slightly on the pressure: the elementary-cell volume was decreased just by 8% at a pressure of 17 GPa.

The present paper gives results of an x-ray structural study of crystals discovered in the products of explosion of a mixture of gibbsite with hexogen [14]. Using an MBS-9 microscope, several green single crystals with a hexagonal prismatic habitus and one light blue single crystal having the shape of an elongated prism (a maximum size of 0.25 mm, a minimum size of 0.1 mm) were identified among the condensed products of the explosion. Crystals were sampled by the Laue method in an RKOP chamber. The researchers were able to isolate two single crystals of green and light blue color. The crystals were then studied on an SAD-4 automatic diffractometer (MoK $_{\alpha}$  radiation, graphite monochromator) using standard methods. Autoindexing by 25 reflections made it possible to distinguish for the blue crystal a triclinic cell with the parameters  $a = 3.084(1)$  Å,  $b = 8.146(3)$  Å,  $c = 15.411(3)$  Å,  $\alpha = 79.13(2)^{\circ}$ ,  $\beta = 84.33(2)^{\circ}$ ,  $\gamma = 79.14(2)^{\circ}$ ,  $V = 372.58(3)$  Å<sup>3</sup>, and for the second (green) crystal, a hexagonal cell that was one-third in volume. The hexagonal cell had the same parameters and Fedorov group as  $\alpha$ -SiC(6H):  $a = 3.080(1)$  Å,  $c = 15.118(2)$  Å,  $V = 124.20(3)$  Å<sup>3</sup>, Fedorov group  $P6_3mc$ .

The deciphering of the structures of these crystals (divergence factor  $R = 2.5\%$ ) revealed that the crystals discovered in the condensed explosion products were silicon carbide. The green crystals belong to the polytype  $\alpha$ -SiC(6H), and the light blue crystal is a previously unknown modification of SiC.

The gibbsite used in [14] contained 0.6 wt.% silicon dioxide. Presumably, in the explosion silicon dioxide reacted with the carbon that, along with CO<sub>2</sub>, CO, H<sub>2</sub>O, H<sub>2</sub>, and N<sub>2</sub>, is formed on decomposition of hexogen [14]. The green tint of  $\alpha$ -SiC(6H) presumably appeared due to the presence of nitrogen in the lattice, and the light blue tint appeared due to isomorphic substitution of aluminum for silicon [6].

The triclinic cell obtained in the auto indexing can be transformed into a base-centered monoclinic Bravais cell with a larger (twice as large) volume. The transformation of the axes can be expressed by the following vector formulas:

$$\vec{A}_m = -\vec{a}_{tr} + 2\vec{b}_{tr};$$

$$\vec{B}_m = -\vec{a}_{tr};$$

$$\vec{C}_m = -\vec{b}_{tr} + 2\vec{c}_{tr},$$

where  $\vec{a}_{tr}$ ,  $\vec{b}_{tr}$ , and  $\vec{c}_{tr}$  are unit translation vectors along the coordinate axes of the triclinic lattice;  $\vec{A}_m$ ,  $\vec{B}_m$ , and  $\vec{C}_m$  are the same along the axes of the monoclinic lattice.

The axis transformation matrix from the triclinic cell to the monoclinic ( $M_{tr \rightarrow m}$ ) is

$$\begin{vmatrix} \bar{1} & 2 & 0 \\ \bar{1} & 0 & 0 \\ 0 & \bar{1} & 1 \end{vmatrix}.$$

The parameters  $\vec{A}_m$ ,  $\vec{B}_m$ , and  $\vec{C}_m$  are equal to  $A = 16.000$  Å,  $B = 3.084$  Å,  $C = 16.016$  Å, and the angle between the axes  $\vec{A}_m$  and  $\vec{C}_m$  is  $\beta = 109.45^{\circ}$ . The elementary-cell volume is  $V = 745.2$  Å<sup>3</sup>.

The angle  $\beta$  is twice the angle between the symmetry axes of the third and fourth order in the cubic lattice. At the same time, the monoclinic angle coincides with the angle between adjacent edges of the primitive cell of the body-centered cubic lattice.

It is possible to pass from the monoclinic base-centered cell to a primitive rhombic one with half the volume by the following axis transformation:

$$\vec{A}_r = \vec{B}_m;$$

$$\vec{B}_r = \frac{1}{2}\vec{A}_m + \frac{1}{2}\vec{C}_m;$$

$$\vec{C}_r = -\frac{1}{2}\vec{A}_m + \frac{1}{2}\vec{C}_m.$$

The axis transformation matrix  $M_{m \rightarrow r}$  is

$$\begin{vmatrix} 0 & 1 & 0 \\ 1/2 & 0 & 1/2 \\ 1/2 & 0 & 1/2 \end{vmatrix}.$$

The parameters  $\vec{A}_r$ ,  $\vec{B}_r$ ,  $\vec{C}_r$  are  $A = 3.084$  Å,  $B = 9.245$  (3  $\times$  3.082) Å,  $C = 13.069$  (3  $\times$  4.356) Å, and  $V = 372.13$  Å<sup>3</sup>.

With  $Z = 18$ , the calculated density is  $3.220 \text{ g/cm}^3$ , which coincides with the density of the known modifications of silicon carbide [2, 3, 6].

The matrix of direct axis transformation from the triclinic cell to the rhombic one can be obtained from the product of matrices  $M_{m \rightarrow r} M_{tr \rightarrow m}$ :

$$\begin{vmatrix} 0 & 1 & 0 \\ 1/2 & 0 & 1/2 \\ 1/2 & 0 & 1/2 \end{vmatrix} \times \begin{vmatrix} \bar{1} & 2 & 0 \\ \bar{1} & 0 & 0 \\ 0 & \bar{1} & 1 \end{vmatrix} = \begin{vmatrix} \bar{1} & 0 & 0 \\ 1/2 & 1/2 & 1/2 \\ 1/2 & 3/2 & 1/2 \end{vmatrix}.$$

The rhombic-lattice axes are expressed in terms of the triclinic-lattice axes by the vector formulas

$$\vec{A}_r = -\vec{a}_{tr};$$

$$\vec{B}_r = -\frac{1}{2}\vec{a}_{tr} + \frac{1}{2}\vec{b}_{tr} + \frac{1}{2}\vec{c}_{tr};$$

$$\vec{C}_r = \frac{1}{2}\vec{a}_{tr} + \frac{3}{2}\vec{b}_{tr} + \frac{1}{2}\vec{c}_{tr}.$$

The rhombic-lattice parameters  $a$ ,  $b$ , and  $c$  are related to the parameter  $a$  of the hexagonal and cubic lattices of silicon carbide in the following way:  $a_r = a_h$ ,  $b_r = 3a_h$ ,  $c_r = 3a_c$ . The blue single crystal discovered in the condensed explosion products presumably belongs to a previously unknown modification of silicon carbide. The new modification of silicon carbide is denoted  $\gamma$ -SiC.

According to E. S. Fedorov's law of crystallographic limits, the world of crystals is not single, but is sharply divided into two kingdoms called types: cubic and hexagonal [7].

The remaining crystals, according to E. S. Fedorov's theory of parallelhedra, are derived from these two types by slight deformations and shifts. The result of homogeneous deformations can be described using the axis transformation matrix. The  $\gamma$ -SiC lattice can be regarded as a distorted  $\beta$ -SiC lattice. The vectors  $\vec{A}_r$ ,  $\vec{B}_r$ , and  $\vec{C}_r$  of the rhombic lattice of  $\gamma$ -SiC are related to the vectors  $\vec{a}_c$ ,  $\vec{b}_c$ , and  $\vec{c}_c$  of the cubic lattice by the following relationships:

$$\vec{A}_r = -\frac{1}{2}\vec{a}_c + \frac{1}{2}\vec{b}_c + 3\vec{c}_c;$$

$$\vec{B}_r = \frac{1}{2}\vec{a}_c + \frac{5}{2}\vec{b}_c;$$

$$\vec{C}_r = \frac{1}{2}\vec{a}_c - \frac{1}{2}\vec{b}_c.$$

Thus, the transformation from the cubic lattice of  $\beta$ -SiC

to the rhombic lattice of  $\gamma$ -SiC is described by a matrix of the following form:

$$M_{c \rightarrow r} = \begin{vmatrix} \bar{1}/2 & 1/2 & 3 \\ 1/2 & 5/2 & 0 \\ 1/2 & \bar{1}/2 & 0 \end{vmatrix}.$$

The absolute values of the parameters  $\vec{A}_r$ ,  $\vec{B}_r$ , and  $\vec{C}_r$  (according to the matrix  $M_{c \rightarrow r}$  for  $a_c = 4.359 \text{ \AA}$ ) are as follows:  $A = 3.082 \text{ \AA}$ ,  $B = 9.247 \text{ \AA}$ ,  $C = 13.077 \text{ \AA}$ , and  $V = 372.7 \text{ \AA}^3$ . The calculated values of the rhombic-lattice parameters virtually coincide with the data on single crystals.

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